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(54) Microfiber and its manufacturing method

(57) This super micro-fiber is mainly prepared by the materials of polyamide (component A) and copolyester (component B). Because these two polymers have poor affinity, a little bit of the third component (component C) is added thereto so as to improve their affinity. The component B is polyethylene terephthalate with the additives of straight-chained fatty di-acid or di-ol or isophthalic acid 5~40% mol while polymerizing to decrease its melting point and melting viscosity; however, its molecular weight is appropriately maintained. The component C contains the 5-sodium sulfodimethyl-isophthalate 0~1% mol besides the additives in the component B. Meanwhile, the melting viscosity ratio between component A and B in the operation temperature should be controlled in 4~10. The melting viscosity of

component C should be kept between those of component A and B. The composition of component A is 30~50 % wt, component B is 40~65 % wt and component C is 5~10 % wt. The three components are blended with a single- or twin-screw extruder. The blended polymer which has the structure of polyamide(island)/ polyester(sea) is spun with spinneret pack pressure 70~130 kg/cm² and taking up velocity 600~1500 m/min. The spun fiber can be drawn furthermore. The copolyester will be removed by dissolving the fiber in alkaline solution and the remained is the polyamide super micro-fiber of 0.0003~0.003 dpf. This manufacturing method can be applied in the production of artificial leather with meticulous feeling.

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Description

Field of the invention

[0001] The present invention is to blend polyamide and polyethylene terephthalate by an extruder. The polyamide becomes discontinuous island phase and polyethylene terephthalate becomes continuous sea phase thereby. Through spinning or further drawing, the polyethylene terephthalate is resolved and removed and polyamide micro-fiber is formed. The dimension of polyamide micro-fiber can reach 0.0003~0.003 dpf (denier per filament). The main skill of the present invention is to apply the modification and the viscosity control of polyethylene terephthalate, and the blended composition so as to cause the polymer composition to form a steady island structure.

Description of prior art

[0002] As disclosed in patent publication No. 177413 and 168750 of the R.O.C., the present process of manufacturing super micro-fiber is to use polyester and polyamide as raw materials. Utilize a special spinneret plate design such as sea island shape or radius shape to proceed complex spinning. Melt and remove one polymer therein so as to produce micro-fiber. Due to the limitation of island arrangement of the said process, a single orifice can generally have 37 partings at most so that the dimension of micro-fiber can only reach 0.025 dpf or so.

[0003] In order to obtain much thinner fiber, Japan patent laid-open No. 40-9429, 41-7886, 41-7893, and 51-6261 disclose to blend polyamide or polyester with polyethylene (PE), polypropylene (PP), or polystyrene (PS) granules. After spinning, the continuous phase is melted and removed by solvent so as to obtain super micro-fiber. The skill has been applied to the production of artificial leather. The solvent treatment is, however, a serious problem. For example, Xylene and Toluene, which are used to dissolve PE and PP, are hazardous to human and are expensive as well. Consequently, they are not recommended.

[0004] Furthermore, polyamide and polyethylene terephthalate are mass produced to be used as raw materials of fibers; it is considered to use polyamide and polyethylene terephthalate blending method in order to develop super micro-fiber. However, due to poor affinity between polyamide and polyethylene terephthalate, it is difficult to blend and spin. Japan patent No. 特開平 8-27626 discloses a method that polyamide and polyethylene terephthalate are blended and then spun at high temperature for quite a long time so as to produce super micro-fiber of polyamide. An exchange reaction is proceeded in between polyamide and polyethylene terephthalate under a high temperature and proceeded for a long time so as to increase the affinity between these two polymers. However, the processing time of

blending and spinning in general practice is not long enough. In addition, a serious pyrolysis happens to polymers from time to time when operated at a high temperature, which affects the spin character and the quality of yarn.

Detailed description of the present invention

[0005] In order to produce 0.0003~0.003 dpf polyamide super micro-fiber by utilizing a simple and economical equipment, meanwhile to solve the problem of solvents' treatment and to find an application route to consume polyamide and polyester which have been mass-produced, the present invention creates an improved method.

[0006] The present invention is a manufacturing method of super micro-fiber. The dimension of polyamide fiber obtained by this method can reach 0.0003~0.003 dpf. This method uses polyamide (component A) 30~50 % wt, copolyester (component B) 40~75 % wt, and copolyester (component C) 5~10 % wt as raw materials. The mixed granules of these three components A, B, and C are further well blended by utilizing a single- or twin-screw extruder. The polyamide in the blended polymer becomes island phase; the copolyester (component B) becomes sea phase; the copolyester (component C) spreads between component A and B as an affinity improver. After granulation and then spinning or directly spinning and drawing processing, the polyester is resolved and removed so as to form 0.0003~0.003 dpf polyamide super micro-fiber.

[0007] The copolyester (component B) used in the present invention is terephthalic acid and ethylene glycol to which the straight-chained fatty di-ol or di-acid, or isophthalic acid are added while polymerizing. The addition ratio is 5~40 % mol so as to decrease melting point and to decrease the melting viscosity of polymer during processing while the molecular weight is free from too low so as to assure the strength of spun and processed fiber. Therefore, the IV (Intrinsic Viscosity) thereof should be greater than 0.5. If the said copolyester is greater than 40 % mol, the polymer is apt to suffer phase separation; meanwhile, too low of melting point may cause the polymer being difficult to crystalize and cause the fiber to adhere each other during processing. Lower than 5 % mol can cause difficulty to decrease melting viscosity.

[0008] The copolyester (component C) used in the present invention is terephthalic acid and ethylene glycol to which not only those additives for component B to lower melting point but also 5-sodium sulfodimethylisophthalate 0~1 % mol are added. Component C serves as an improver of the affinity between component A and B: wherein sulfonic acid group and polarized polyamide absorb each other so as to cause the improvement of affinity. When 5-sodium sulfodimethylisophthalate is more than 1 % mol, the radius of formed island and pitch of island are too small; consequently,

the sea phase is difficult to melt and remove.

[0009] In order to obtain a steady polyamide sea phase by controlling the three polymer components A, B, and C after being blended, it is inevitable to select appropriate weight composition and melting viscosity ratio of component A, B, and C. In general, the lower component is apt to become island phase, and the component with higher viscosity is as well apt to become island phase. In the present invention, it is wanted that the component of island be increased in order to decrease the loss when melting and removing and also the loss of meticulous feeling of fiber thereafter. Therefore, the melting viscosity ratio of island phase should be raised as compensation. On the other hand, when the material characteristics of polyamide which is in fiber phase should be kept in good condition, it is inevitable to use the copolyester owning a low melting viscosity so as to reach the needed goal. However, when the copolyester granule which owns a low melting viscosity is used as a raw material of blending, there is often a limitation of cutting owing to the viscosity being too low. Due to the said limitation, the melting viscosity ratio of component A and B under operation temperature can only be kept in 4~10. Consequently, the weight of component A should be 30~50 % wt of total weight so as to have polyamide become steady island phase. while component A weighs over 50 % wt, there may happen unexpected consequences that sea exists in island, and island exists in sea, or even component A occurs phase inversion into sea. On the other hand, while component A weighs under 30 % wt, the feeling of thickness of leather is less than expected. The weight of component C -- copolyester should be 5~10% wt of total weight. While component C weighs over 10 % wt, the island diameter and island pitch will be too small ; as a result, copolyester is difficult to completely melt and remove. On the other hand, while component C weighs less than 5 % wt, component C will be difficult to even distribute between component A and B; consequently, the improved affinity is not enough. Meanwhile, the melting viscosity of component C is controlled between those of component A and B so as to contribute to the distribution of component C between A and B. Based on those mentioned above, component B -- copolyester weighs 40~65% wt of total weight.

[0010] While the component A, B, and C are being spun after being blended, the spinneret pack pressure should be kept in 70~130 kg/cm². When the said pressure is over 130 kg/cm², the structure of sea and island is apt to change so as to occur uneven thickness and easy break-off while spinning. On the other hand, when the said pressure is less than 70 kg/cm², the viscosity will be too low and the fiber will be broken off due to the lack of strength. The taking up velocity being kept in 600~1500 m/min is most appropriate since the strength of blended and spun fiber is lower than single polymer.

[0011] Analysis method of IV (Intrinsic viscosity) : The IV value of the present invention uses Phenol/m-cresol

=2/3 as solvent and polymer concentration 0.5g/100ml solution; which is analyzed at 25 °C.

[0012] To let the examiner further understand characteristics of the manufacturing method of the present invention, examples are explained as follows:

Example I:

[0013] Nylon 6, whose RV (relative viscosity) is equal to 2.47 (measured when 1 % wt in 95 % sulfuric acid), weighs 50 % wt. Copolyester (component B) is polyethylene terephthalate (PET) which contains adipic acid 13 % mol. IV is equal to 0.508. Melting viscosity is 365 poise at 270°C and weight is 45 % wt. Copolyester (component C) is PET which contains isophthalic acid 10 % mol and 5-sodium sulfodimethylisophthalate 0.3 % mol. IV is equal to 0.503 and weight is 5 % wt. These three granules are well mixed in a rolling barrel mixer and dried; then melted and blended by a twin screw extruder (L/D = 36, D = 37mm) at temperature 270 °C. The blended granules are dried by N₂ at 130 °C for 8 hours and spun at a temperature of 265 °C thereafter. The spinneret pack pressure is 74 kg/cm². The amount of holes of spinneret plate is 484. The L/D of spinneret hole is 0.5 mm/ 0.3 mm. The taking-up velocity is 800 m/min. The dimension of fiber during the first spinning is 13 denier. Drawn at a draw ratio of 3.0 thereafter. The polyester is completely melted and removed by NaOH 5 % wt at 110 °C for 60 min. Then, analyze 0.0014~0.003 dpf Nylon 6 fiber by using Hitachi S-520 scan electrical microscope. The result is shown in attached figure (enlarged ratio is 5000).

Example II:

[0014] Nylon 66, whose RV (relative viscosity) is equal to 2.50, weighs 50 % wt. Copolyester (component B) is polyethylene terephthalate (PET) which contains adipic acid 10 % mol. IV is equal to 0.512 and weight component is 40 % wt. Copolyester (component C) is PET which contains isophthalic acid 10 % mol and 5-sodium sulfodimethylisophthalate 0.3 % mol. IV is equal to 0.503 and weight is 10 % wt. The blending and spinning conditions are the same with those of Practicing example I. The spinneret pack pressure is 120 kg/cm². The taking-up velocity is 600 m/min. The polyester is completely melted and removed. The dimension of Nylon 66 micro-fiber is 0.0035 ~ 0.0014 dpf.

Claims

1. A super micro-fiber and its manufacturing method, which uses 30 ~ 50 % wt polyamide (component A) and 40 ~ 65 % wt copolyester (component B) as main materials with the addition of 5 ~ 10 % wt copolyester (component C) as an improver of affinity between component A and B. After being

blended by a single- or twin-screw extruder, polyamide becomes island phase and copolyester becomes sea phase. The melting viscosity ratio of component A and B, at operation temperature, is 4 ~ 10, and the melting viscosity of component C is controlled between those of component A and B. Through spinning following granulation or directly spinning and then drawing processing, the polyester is melted and removed so as to form 0.0003 ~ 0.003 dpf polyamide micro-fiber.

2. A super micro-fiber and its manufacturing method as defined in Claim 1, wherein copolyester (component B) is terephthalic acid and ethylene glycol with the additives of straight-chained fatty diol or di-acid or isophthalic acid while polymerizing. The addition ratio is 5 ~ 40 % mol so as to decrease melting point and control its IV > 0.5
3. A super micro-fiber and its manufacturing method as defined in Claim 1, wherein copolyester (component C) is terephthalic acid and ethylene glycol with those additives described in Claim 2 so as to decrease melting point plus the addition of 5-sodium sulfodimethylisophthalate 0 ~ 1 % mol as an improver of affinity between component A and B.
4. A super micro-fiber and its manufacturing method as defined in Claim 1, wherein the blended polymer as per its manufacturing method should be spun at a spinneret pack pressure of 70 ~ 130 kg/cm² and at a taking-up velocity of 600 ~ 1500 m/min.





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EUROPEAN SEARCH REPORT

Application Number
EP 01 10 6451

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.7)
A	GB 1 432 228 A (ASAHI CHEMICAL IND) 14 April 1976 (1976-04-14) * the whole document *	1-4	D01F6/60 D01F6/90 D01F6/92 D01F8/12 D01F8/14
A	DATABASE WPI Section Ch, Week 198131 Derwent Publications Ltd., London, GB; Class A23, AN 1981-56235D XP002176323 & JP 56 073115 A (UNITIKA LTD), 17 June 1981 (1981-06-17) * abstract *	1-4	
A	DATABASE WPI Section Ch, Week 199305 Derwent Publications Ltd., London, GB; Class A23, AN 1993-042835 XP002176324 & KR 9 203 139 B (DONG YANG NYLON CO), 20 April 1992 (1992-04-20) * abstract *	1-4	
			TECHNICAL FIELDS SEARCHED (Int.Cl.7)
			D01F
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 30 August 2001	Examiner Tarrida Torrell, J
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**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.**

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This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on
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30-08-2001

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
GB 1432228 A	14-04-1976	JP 893828 C	18-01-1978
		JP 49005888 A	19-01-1974
		JP 52019195 B	26-05-1977
		CA 1000683 A	30-11-1976
		DE 2318831 A	31-10-1973
		FR 2180059 A	23-11-1973
		US 3936394 A	03-02-1976
JP 56073115 A	17-06-1981	NONE	
KR 9203139 B	20-04-1992	NONE	

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